

CHAPTER IV

BLEND OF LOW-DENSITY POLYETHYLENE WITH NYLON COMPATIBILIZED WITH SODIUM-NEUTRALIZED CARBOXYLATE IONOMERS: PHASE MORPHOLOGY AND MECHANICAL PROPERTIES

ABSTRACT: The effectiveness of an ethylene-methacrylic acid copolymer partially neutralized with sodium (Na-EMAA) as a compatibilizer for blends of low-density polyethylene (LDPE) with polyamide 6 (PA6) was investigated. The phase morphology and mechanical properties of these blends were investigated over a range of compositions, using scanning electron microscopy (SEM), tensile testing, impact testing, dynamic mechanical analyzer (DMA) and Shore D hardness. The percentage water absorption of uncompatibilized blends decreased with increasing LDPE content. PA6/LDPE blends showed reduced mechanical properties after mixing versus a simple mixing rule, attributed to phase separation between the two polymers. SEM micrographs showed that the addition of a small amount of Na-EMAA ionomer improved the compatibility of PA6/LDPE blends as evidenced by a reduction in dispersed phase size from 15 μm to 1.5 μm ; this reduction was achieved at a Na-EMAA level of 0.5% and did not increase with higher amounts of ionomer. The mechanical properties of compatibilized blends improved as compared with uncompatibilized blends. Moreover, the shifting of loss modulus peaks in DMA results of blends containing Na-EMAA ionomer, indicated that there are some improvements in the compatibility of resulting blends. During blending, chemical and/or physical reactions had taken place between PA6 and Na-EMAA ionomer.

Key words: sodium-neutralized carboxylate; ionomers blend; compatibilizer; low-density polyethylene; nylon6

INTRODUCTION

One way to develop new polymeric materials is to blend two homogeneous polymers. Some pairs of polymers are miscible and lead to single-phase homogeneous systems, such as polyethylene/polystyrene blends (PE/PS), or polycarbonate/polyethylene terephthalate blends (PC/PET), but most polymer blends are thermodynamically immiscible with each other and on mixing form multiphase morphologies. The structures obtained are usually thermodynamically unstable, and the mechanical properties of the blends are poor because of the lack of adhesion between the phases (Gadekar *et al.*, 1997; Leewajanakul *et al.*, 2003). The morphological features of an incompatible blend, such as the size of the dispersed phase domains and the adhesion at the interface, play an important role in determining its mechanical properties (Gadekar *et al.*, 1997; Lahor *et al.*, 2004). Evidence from the open literature suggests that the blend morphology can be altered by adding a compatibilizer; the mechanism of how this compatibilizer works can vary. In the case of the ionomeric compatibilizer that is the focus of this report, the ionomer reacts with one component of the blend (PA6) and is believed to interact with the other component (LDPE) because of the similarity of the backbones.

Polyolefins and polyamides are two important classes of polymers. Due to low cost, lightweight, high strength, high barrier properties to moisture and ease of processing, LDPE is an ideal material for film and container applications. However, PA/PE blends are immiscible due to the presence of polar groups in the PA and the non-polar group ones of the PE. Blend properties can be improved using a suitable compatibilizer such as ethylene methacrylic acid copolymer (Scaffaro *et al.*, 2003) acrylates such as LDPE-g-butyl acrylate (Raval *et al.*, 1991) and ionomers such as zinc-neutralized ethylene-methacrylic acid ionomers (Armat and Moet, 1993; Leewajanakul *et al.*, 2003)

The functionalization of polyethylene with a small amount of ionic groups is a particular attractive way of compatibilizing polyamide with polyethylene, because the amide groups may interact with the ionomer via hydrogen bonding, ion-dipole interactions, or/and metal ion coordination during melt blending. In addition, the amide groups, or the primary amine that terminates the chain, may react with the carboxylic acid on the ionomer. Much less studied has been the interaction between

LDPE and the ionomer, the two materials are not totally miscible but are believed to interact with one another because of the similarities of the polymer backbones. The introduction of these specific interactions improves the compatibility and may promote miscibility of polyamide and polyethylene blends.

Ionomers contain both nonionic repeat units, and a small amount of ion containing repeat units. Surlyn[®] is a commercial thermoplastic ionomer resin that was introduced by DuPont in the early 1960's. Surlyn[®] is a random copolymer poly(ethylene-co-methacrylic acid) (EMAA) and is produced through the copolymerization of ethylene and methacrylic acid via high-pressure free radical reaction, similar to that used for the production of LDPE. The incorporation of methacrylic acid units can be neutralized with suitable cation, commonly either zinc (Zn^{2+}) or sodium (Na^+). Commercially, the fraction of acid groups that are neutralized is typically between 20 and 60%. There are significant differences between zinc and sodium ionomers, primarily due to the difference in the aggregate structure for the transition metal zinc and the alkali metal sodium. Zinc ionomers are softer, clearer and show adhesion to metal surfaces.

A few mol% of ionic groups along the backbone of the ionomer has a tremendous effect upon the morphology and the properties of the polymer. The presence of the methacrylic acid units and the neutralized carboxylate anion/cation pairs provides sites for ionic interactions. Interactions between ion pairs, and the nonpolar nature of the backbone cause the ions to aggregate together and act as "thermally reversible crosslinks". At room temperature, the materials have some characteristics of crosslinked materials, such as the ability. When heated, the ionic groups will lose their attractions for each other and the chains will be able to move around freely, although the viscosity is typically much higher than the unneutralized copolymer. Surlyn[®] ionomers have excellent heat sealability, hot tack and they can be sealed through contaminants, therefore they are used as a premium heatseal layer for packaging films, especially recommended for high speed packaging machines.

Previous studies have shown that sodium-neutralized ethylene-co-methacrylic acid ionomer is an effective compatibilizer in polyamide6/low density polyethylene blends (Lahor *et al.*, 2004). The addition of this ionomer to PA6/LDPE

blends resulted in a significant decrease in the dispersed phase size and increase in thermal behavior of the resulting blends.

The aim of this work is to study the effect of compatibilizer content of polyamide6/low density polyethylene blends using sodium-neutralized ionomer (Na-EMAA) as a compatibilizer. Attention will be focused on the mechanical properties and phase morphology of the resulting blends.

EXPERIMENTAL

Materials

Ultramid B3 PA6 (density 1.31 g/cm³) was supplied by BASF (Thailand) Co.,Ltd. Low-Density Polyethylene, LD 1450J, was an injection molding grade polymer (density 0.914 g/cm³) graciously supplied by Thai Polyethylene Co.,Ltd. Sodium-neutralized poly(ethylene-co-methacrylic acid) ionomer marketed under the trademark Surlyn[®] 8527 (density 0.94 g/cm³) was supplied by DuPont (USA).

Blends Preparation

Prior to blending, all the blend compositions were externally mixed using a tumble mixer for 10 minutes followed by drying under vacuum at 60°C for 24 hours. The materials were blended in a Collin D-8017 T-20 twin-screw extruder using a screw speed of 40 rpm. The blends were extruded through a single strand die, the extrudates were cooled in a water bath, then dried at ambient temperature and pelletized.

Specimen Preparation

Test specimens were prepared using a Wabash V 50 H 50-ton compression-molding machine. The obtained pellets were placed in a picture frame mold and the mold was preheated at 240°C for 3 minutes in the press without any applied pressure

for complete melting. The mold was then compressed under a force of 10 tons for a further 3 minutes after which the mold is cooled to 40°C under pressure. Cooling times were on the order of 5 minutes. Test specimens were cut from the molded sheets using a pneumatic die cutter.

Scanning Electron Microscopic Analysis

The fracture micrographs as well as the dispersed structure of the fractured samples were studied using a scanning electron microscope, JEOL (MP 152001), operated at 15-25 kV. The samples fractured under liquid nitrogen and were also subjected to selective extraction of the LDPE and Na-EMAA ionomers phases by immersing in hot decalin to remove LDPE or in formic acid to remove the Ny6. The specimens were then coated with gold, under vacuum, to make them electrically conductive. The number average diameter (d_n) was calculated using equation (1),

$$d_n = \frac{\sum(n_i d_i)}{\sum n_i} \quad (1)$$

where; n_i is the number of droplet and d_i is the diameter the i th droplet.

Dynamic Mechanical Analysis

Dynamic mechanical of these blends were studied using a Solid Analyzer RSA II (Rheometric scientific). The storage modulus (G') and loss modulus (G'') were measured as a function of temperature. The film and fiber fixture was used to mount the samples and temperature step of 4 K intervals. All experiments were performed at 10 Hz frequency and 0.05 % strain amplitude using force tracing dynamic force.

Mechanical Properties Testing

Tensile properties, impact property and hardness of the blends were determined from the compressed specimens following the test conditions suggested by ASTM.

An Instron Universal testing machine was used to measure the tensile strength and tensile modulus of the blends. The tests were conducted according to ASTM D638-91 test procedure, using a crosshead speed of $50 \text{ mm}\cdot\text{min}^{-1}$. Izod impact strength was measured using a Zwick Impact tester according to ASTM D 256-92 test procedure with a 2.7 J pendulum. A Shore D durometer was used to measure hardness of the blends. The test was conducted according to ASTM D 2240 test procedure.

All the tests were carried out at room temperature 30°C and the results were obtained from the average of ten specimens for every blend ratios.

The Percentage Water Absorption Measurement

The percentage water absorption measurement was conducted per ASTM D 570. All the specimens used for the study had dimensions of about $5.4 \times 1.8 \times 0.3 \text{ cm}^3$. The test samples were dried at 50°C for 24 h prior to measurement. The samples were first cooled, weighed and the placed in a container of boiling distilled water, supported on edge and entirely immersed in water. After 2 h. the specimens

were removed from water and cooled in distilled water maintained at room temperature. After 15 min the specimens were removed from water, wiped with dry cloth and weighed immediately. The percentage water absorption was determined as follows:

Percentage water absorption =

$$\frac{\text{weight of wet sample} - \text{conditioned weight}}{\text{conditioned weight}} \times 100$$

RESULTS AND DISCUSSION

Phase Morphology

The morphology of different blends was investigated by scanning electron microscope (SEM) on freeze-fracture specimens. SEM micrographs of freeze-fracture surfaces of uncompatibilized blends showed a clear-cut, two phase morphology as shown in Figure 4.1. The presence of dispersed phase, consisting of predominantly spherical droplets imbedded in a matrix, was clearly observed from the micrographs over the whole composition range. The micrographs of compatibilized PA6/LDPE blends are shown in Figures 4.2 and 4.3. The addition of the ionomer resulted in a decrease of the dispersed phase size in both cases, i.e. whether LDPE or PA6 was the dispersed phase. The reduction of dispersed phase size when the compatibilizer was added was due to the ability of Na-EMAA ionomer as the compatibilizer to reduce the interfacial tension between two phases. The number average dispersed phase size ranged between 2 and 5 μm , which were much smaller than in the uncompatibilized blends (Table 4.1). These observations could be caused by the interactions between Na-EMAA ionomer and PA6. Macknight *et al.* and as well as others have suggested that two possible specific interactions between nylon and ionomers are hydrogen bonding between the amide nitrogen on nylon and the carboxylic acid on the ionomers, and a covalent amide bond, which can form between the primary amines at the end of the Nylon chains and the carboxylic acids of the ionomers. This later interactions can be confirmed using the

Molau test, by adding of formic acid to blends (Lahor *et al.*, 2004; Raval *et al.*, 1991). For uncompatibilized blends, a separation of PA6 from LDPE was observed, whereas the PA6/LDPE/Na-EMAA blends gave rise to a colloidal suspension in formic acid. This test suggests that bonds were formed between PA6 and Na-EMAA, and that there was some mixing between the Na-EMAA and the PA6.

Figure 4.4 shows the number average size of dispersed phase of the compatibilized blends. The result showed that approximately 0.5 wt% of Na-EMAA ionomer was sufficient to produce a maximum reduction of the dispersed phase size. No further decrease in phase size was achieved by adding more Na-EMAA ionomer, as seen from the plateau region of the plots between average dispersed phase diameter and amount of Na-EMAA ionomer added (Lahor *et al.*, 2004).

Morphologies of the fractured and etched surfaces of the PA6/Na-EMAA blends are shown in Figure 4.5. An increase of the system homogeneity can be observed with increasing Na-EMAA ionomer content in PA6/Na-EMAA blends which caused a decrease in the size of the dispersed phase and gave a more homogeneous morphology. The reduction in particle size of the dispersed phase due to the chemical reactions taking place between two phases as confirmed by the Molau test.

The fracture surface of blends of LDPE/Na-EMAA appeared to be homogeneous since no evidence of phase separation (see Figure 4.6). The compatibility in LDPE/Na-EMAA could be attributed to the miscibility between polyethylene segments of LDPE and ethylene backbone segments of Na-EMAA. However, one cannot be sure that these two systems are not phase separated, since both components dissolve in the same solution. DSC results show that the crystals at least are phase separated, i.e. there are two melting points, one characteristic to the LDPE and another characteristic to the ionomer (Lahor *et al.* 2004).

Dynamic Mechanical Analysis

Miscibility of polymer blends was studied using Dynamic Mechanical Analysis (DMA). In this study the dynamic mechanical properties of blends were determined as a function of temperature dependence of storage modulus (E') and loss modulus (E''), respectively. The plot of loss modulus (E'') as a function of temperature of pure component is shown in Figure 4.7. The main relaxation temperatures of PA6 were detected at 16.2°C (α relaxation) which is ascribed to the glass transition temperature (T_g) and around -82.7°C (β relaxation). The β relaxation, which is observed at around -82.7°C has been explained on the basis of the rotational motion of water molecules and/or the water/polymer complex (Rupali *et al.*, 1997). The γ relaxation was not found in this test.

The peak of -32.7°C is a transition peak of LDPE which can be compared with those for polyethylene samples. In previous work, the DMA spectrum of polyethylene had three low-temperature peaks, at around -30°C , -78°C and -128°C (Sheng *et al.*, 1999); it is not clear what the molecular motion is that leads to these peaks. For Na-EMAA, the transition peak is much broader, and appears to have shifted to lower temperature with a peak at approximately -41°C . The addition of acid groups would be expected to raise, not lower, the glass transition temperature; so consistent with Sheng *et al.*, this peak does not appear to be due to the glass transition temperature.

Figure 4.8 showed the DMA spectra of the PA6/LDPE blends. The peak at 16.3°C is a T_g of PA6 in the PA6/LDPE blends (PA6 60/ LDPE 40) and -32.5°C is a T_g of LDPE in this blends. Clearly, the T_g 's of PA6 and LDPE in PA6/LDPE uncompatibilized blends did not change as compared with the pure polymer representing further that there are two phases in blends of PA6/LDPE. The addition of Na-EMAA ionomer as a compatibilizer resulted in modification of glass transition temperature (T_g) as can be seen in Figure 4.9. As shown in the loss modulus displayed in Figure 4.10, the transitions for the PA6 and Na-EMAA ionomer shift towards one another, i.e. the lower temperature LDPE transition shifts towards higher temperature, and the higher temperature LDPE transition shifts towards lower

temperature. For example, in the material with a 50/50 composition, the glass transition temperature (T_g) of PA6 shifted toward lower temperature (from 16 °C to 12 °C) while the T_g of Na-EMAA ionomer increased from -41 °C to -24 °C. This results suggest that the mobility of one phase is being affected by the mobility of a different phase, which is consistent with the idea of a chemical reaction of the ionomer with the polyamide, and the mixing of the amorphous sections of the ionomer with the amorphous sections of the polyethylene.

Mechanical Properties

Tensile Properties

Tensile properties of PA6/LDPE blends (without compatibilizer)

The tensile properties of blends between PA6 and LDPE are shown in Figure 4.11 and 4.12. The tensile properties of all uncompatibilized blends at high nylon contents were lower than that pure PA6, including the elongation at break. The addition of a flexible polymer is expected to reduce the tensile strength and modulus, however the reduction in ultimate elongation is likely due to the poor interfacial adhesion between two phases, which resulted in the weak stress transfer from one phase to another phase. However, in the PA6/LDPE blends where LDPE was the major component, the tensile properties were slightly higher than the pure LDPE (Leewajanakul *et al.*, 2003).

Tensile properties of PA6/LDPE blends (with compatibilizer)

Tensile modulus

The tensile properties of compatibilized blends were studied to investigate the effect of Na-EMAA ionomer as a compatibilizer. The increasing in the tensile modulus of PA6/LDPE blends was observed compared to the uncompatibilized blend with the addition of 0.5 wt % Na-EMAA ionomer as a compatibilizer (see Figure

4.13). 0.5 wt% of Na-EMAA also gave the highest the tensile modulus values; which was expected since SEM micrographs suggest that adding more than this amount did not decrease dispersed phase size better.

Tensile strength

Tensile strengths of PA6/LDPE as a function of Na-EMAA ionomer contents are shown in Figure 4.14. In blends containing compatibilizer, the values of tensile strength were lower than that of pure PA6 for all compositions. However, tensile strength of PA6/LDPE blends was enhanced by addition of the Na-EMAA ionomer as a compatibilizer. This indicated that Na-EMAA ionomer improved the interfacial adhesion and reduced the size of the dispersed phase which resulting in better stress transfer between two phases. The effect of compatibilization was more predominant in the 80/20 PA6/LDPE blend composition when compared with other compositions, the tensile strength increased from 37.2 to 40.8 MPa when only 0.5 wt% of Na-EMAA was added. However, the behavior of tensile properties became worse at high compatibilizer content.

Impact Properties

Impact properties of PA6/LDPE blends (without compatibilizer)

Figure 4.15 shows the relationship between impact strength and LDPE content. From the plot it can be clearly seen that the impact strength of PA6/LDPE uncompatibilized blends increased as the LDPE content increased due to the higher impact strength of LDPE.

Impact properties of PA6/LDPE blends (with compatibilizer)

Figure 4.16 shows that the addition of Na-EMAA ionomer caused an improvement in the impact strength of PA6/LDPE blends especially for the 80/20 PA6/LDPE blends. This improvement can also be explained by the improved adhesion between the phases, which allow absorbed energy to transfer from one phase to another phase. Similar results have been reported in Nylon6/VLDPE and Nylon6/PP blends by other work (Gadekar *et al.*, 1997).

Hardness

Hardness of PA6/LDPE blends (without compatibilizer)

The hardness results are shown in Figure 4.17. The hardness of PA6/LDPE binary blends was lower than that pure PA6. Moreover, the hardness of these blends was decreased as the LDPE content increased.

Hardness of PA6/LDPE blends (with compatibilizer)

The PA6/LDPE uncompatibilized blends have lower hardness than PA6/LDPE/Na-EMAA compatibilized blends because of the lack of adhesion between two phases (see Figure 4.18). The hardness of PA6/LDPE blends was enhanced by the addition of small amount of Na-EMAA ionomer to PA6/LDPE binary blends. As with the other measurements, the maximum improvement of hardness was observed when only 0.5 wt % of Na-EMAA ionomer was added to the PA6/LDPE blends.

Water Absorption

The properties of Nylon 6 are very sensitive to humidity, because of absorbed water. Therefore, reducing water absorption would significantly help in the processing of PA6 since the presence of water can lead to molecular weight reductions which in turn lead to property degradation. This problem could possibly be overcome by blending PA6 with LDPE. Figure 4.19 illustrates the percentage of water absorption of the blends as a function of LDPE content, it can be inferred that all the blends have lower percentage water absorption values than that of neat PA6 which is due to the insensitivity of LDPE to moisture thus leading to a significant drop in the percentage water absorption.

Figure 4.20 shows that the percent water absorption of compatibilized blends decreased vs. the uncompatibilized blends. This result is extremely interesting; first since the ionomer will absorb a great deal of water as well; hence the decline is in spite of this fact.

CONCLUSIONS

Results of the study showed that the ethylene-methacrylic acid copolymer partially neutralized with sodium (Na-EMAA) was effective when used as a compatibilizer for blends of LDPE with PA6. SEM micrographs showed that the average size of the dispersed phase decreased significantly by the addition of a small amount of Na-EMAA. Only 0.5 wt % of Na-EMAA was sufficient to produce maximum reduction in dispersed phase size with an average dispersed phase size in the compatibilized blend ranging between 2 and 5 μm . The mechanical properties increased with the addition of a small amount of compatibilizer as well. These phenomena were more pronounced at 80/20 PA6/LDPE with 0.5 wt% of Na-EMAA. DMA data supported that the improvement in the properties of blends containing Na-EMAA ionomer was observed.

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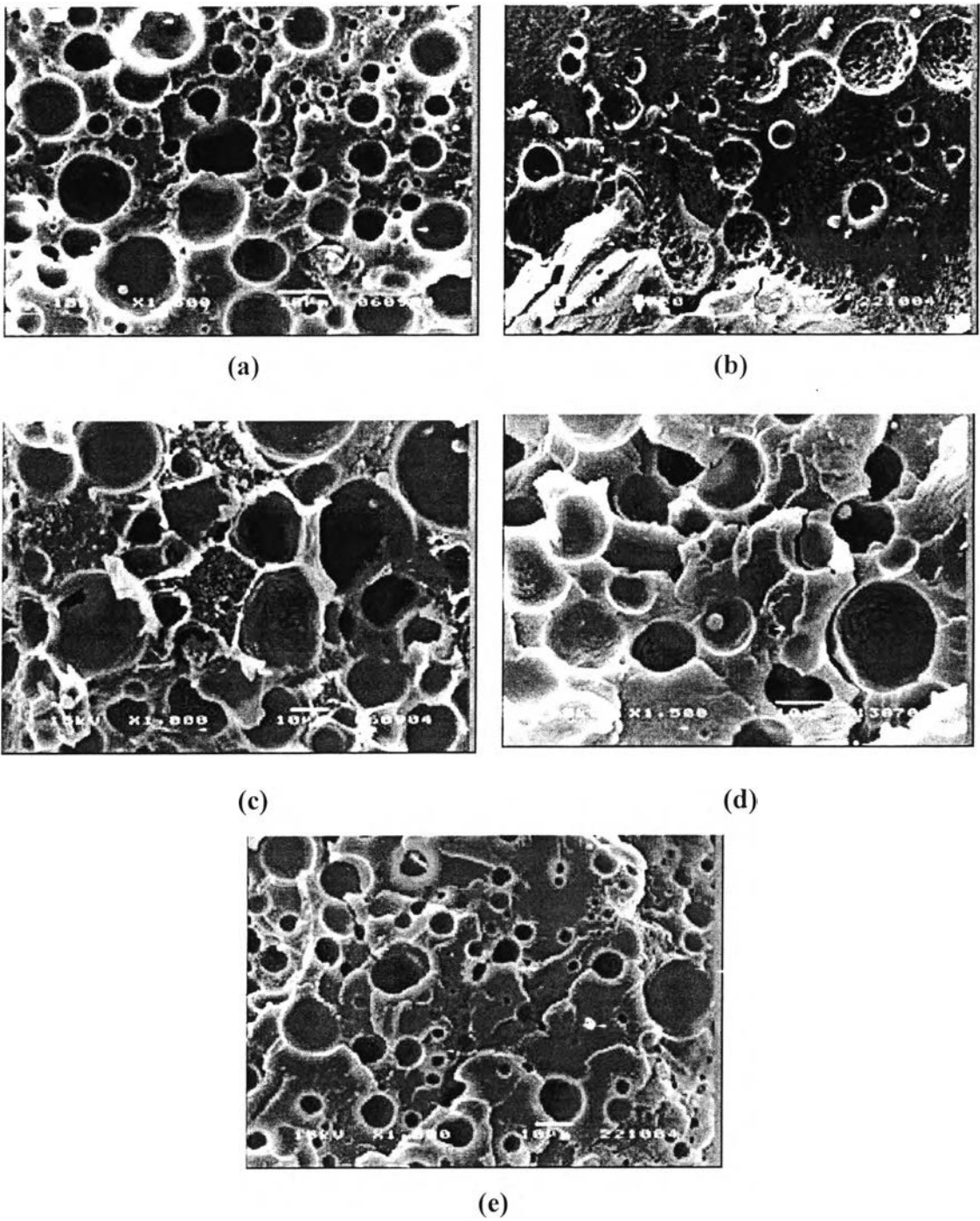


Figure 4.1 The SEM micrographs of blends without Na-EMAA ionomer (Surlyn[®]) as compatibilizer at the following PA6/LDPE ratios: (a) 80/20, (b) 60/40, (c) 50/50, (d) 40/60, (e) 20/80.

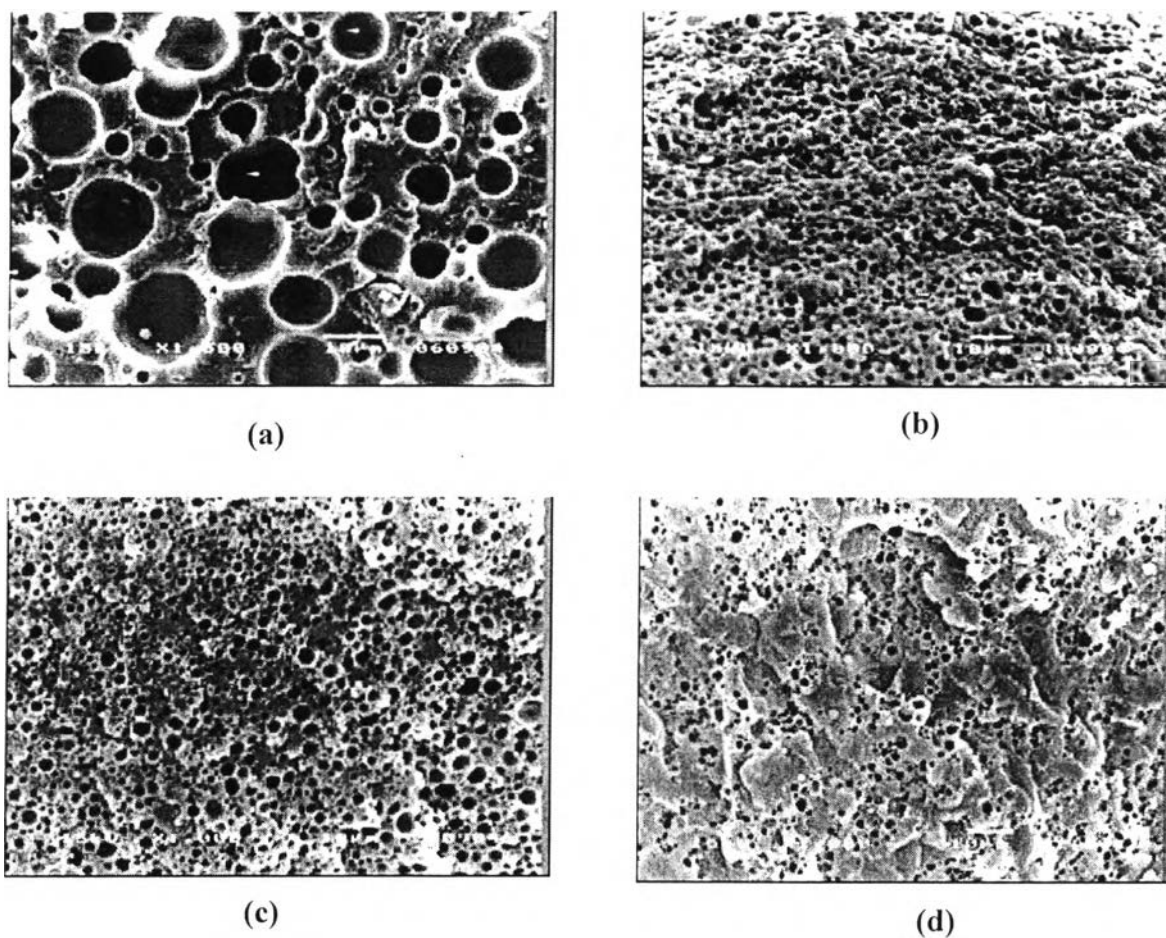


Figure 4.2 The SEM micrographs of 80/20 PA6/LDPE blends with added Na-EMAA ionomer (Surlyn[®]) compatibilizer at the following weight percentages: (a) 0, (b) 0.5, (c) 1.5 and (d) 5.0.

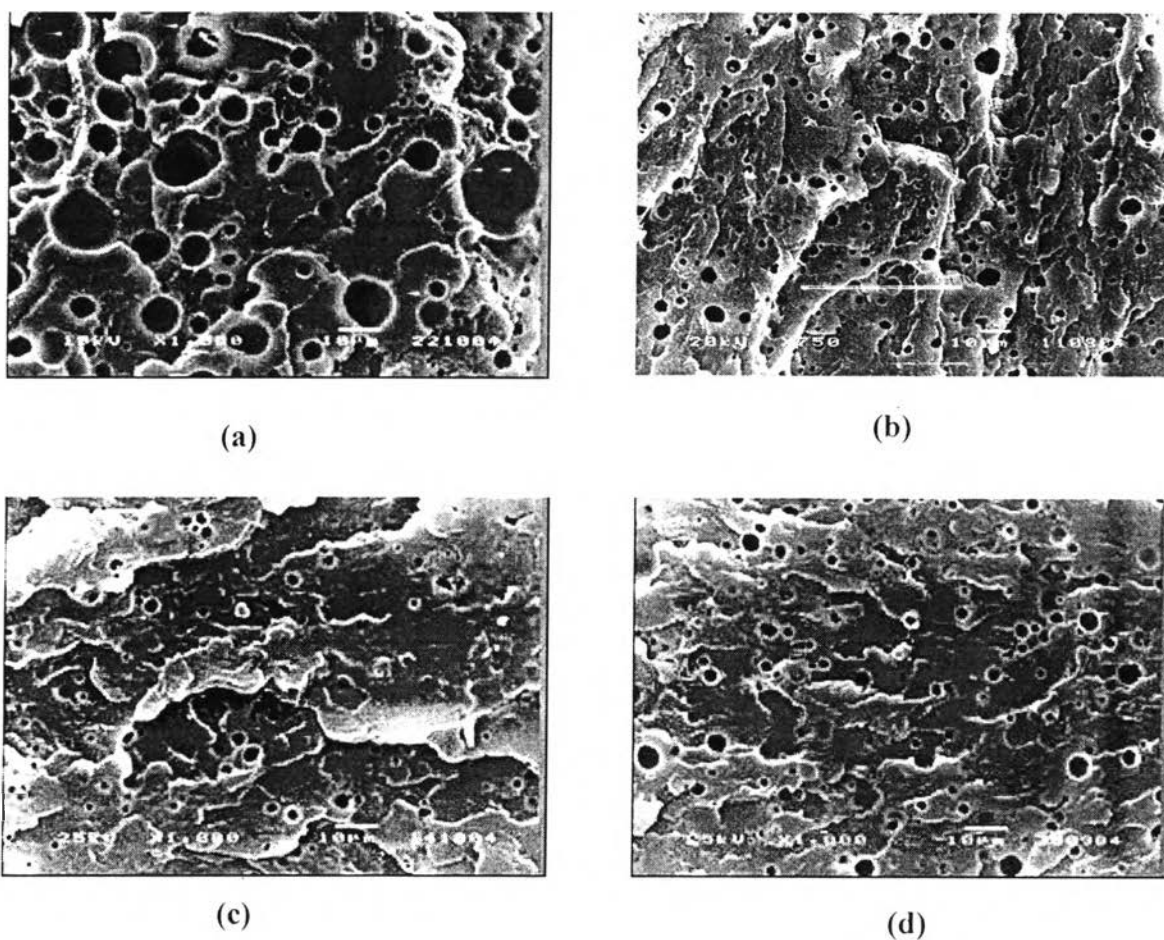


Figure 4.3 The SEM micrographs of 20/80 PA6/LDPE blends with added Na-EMAA ionomer (Surlyn[®]) compatibilizer at the following weight percentages: (a) 0, (b) 0.5, (c) 1.5 and (d) 5.0.

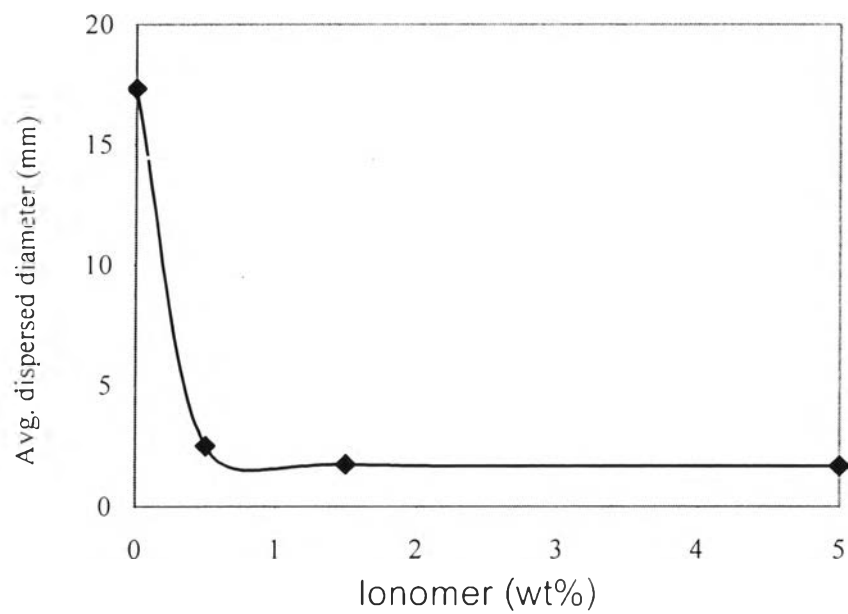


Figure 4.4 The dependence of the number average diameters measured as a function of the Na-EMAA ionomer (Surlyn[®]) content of 80/20 PA6/LDPE blends.

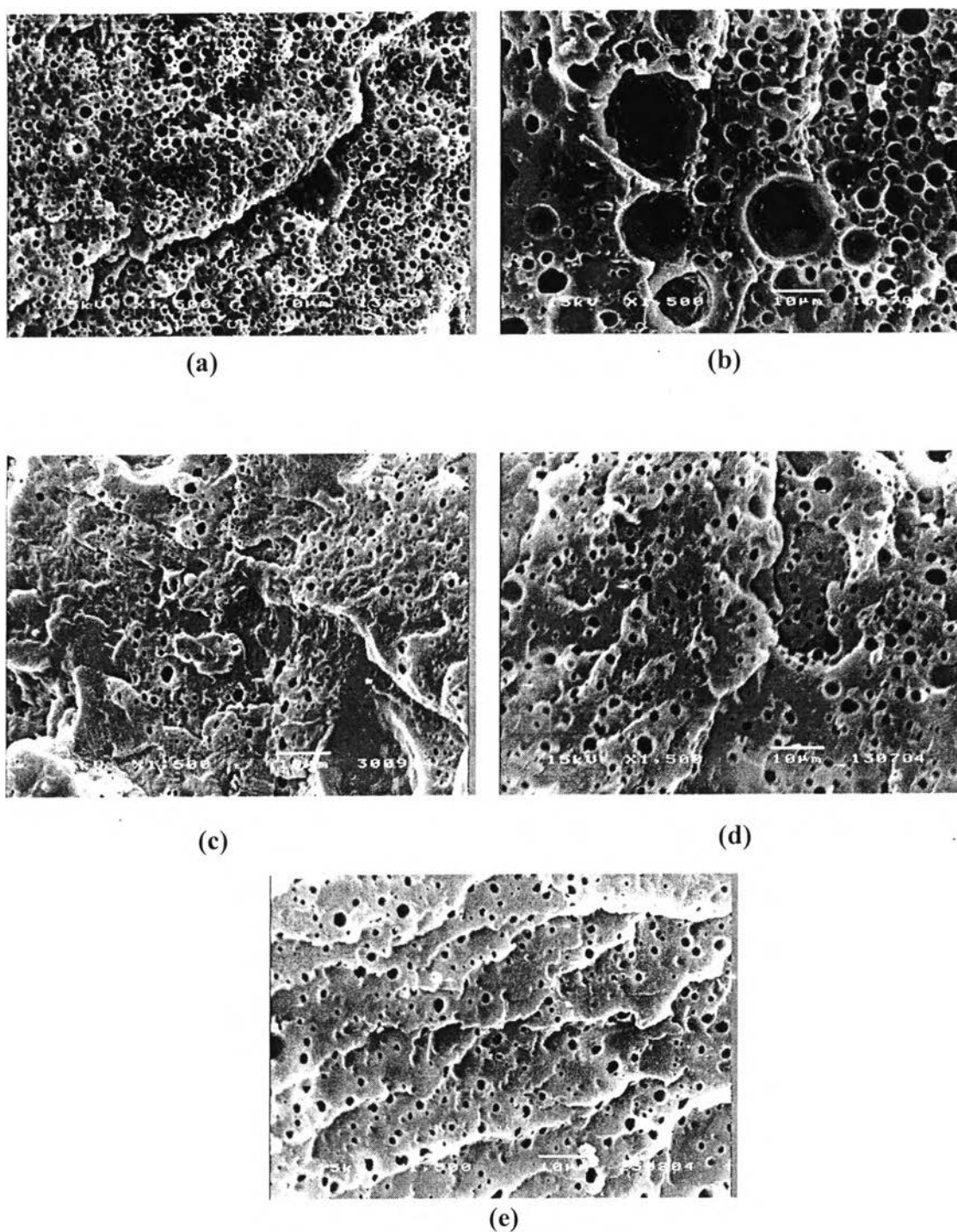


Figure 4.5 The SEM micrographs of blends PA6/Na-EMAA at the following ratios: (a) 80/20, (b) 60/40, (c) 50/50, (d) 40/60, (e) 20/80.

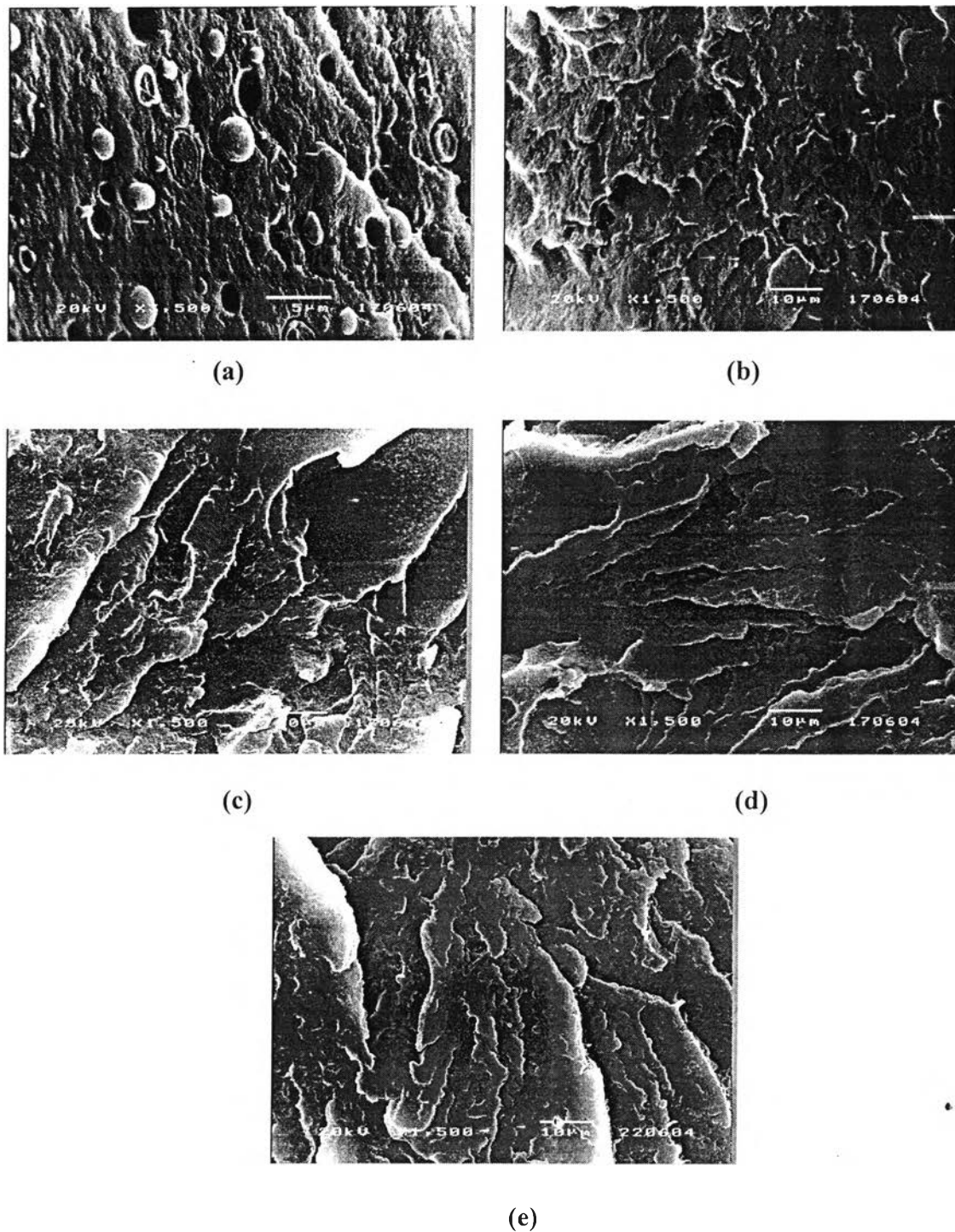


Figure 4.6 The SEM micrographs of blends LDPE/Na-EMAA ionomer at the following ratios: (a) 80/20, (b) 60/40, (c) 50/50, (d) 40/60, (e) 20/80.

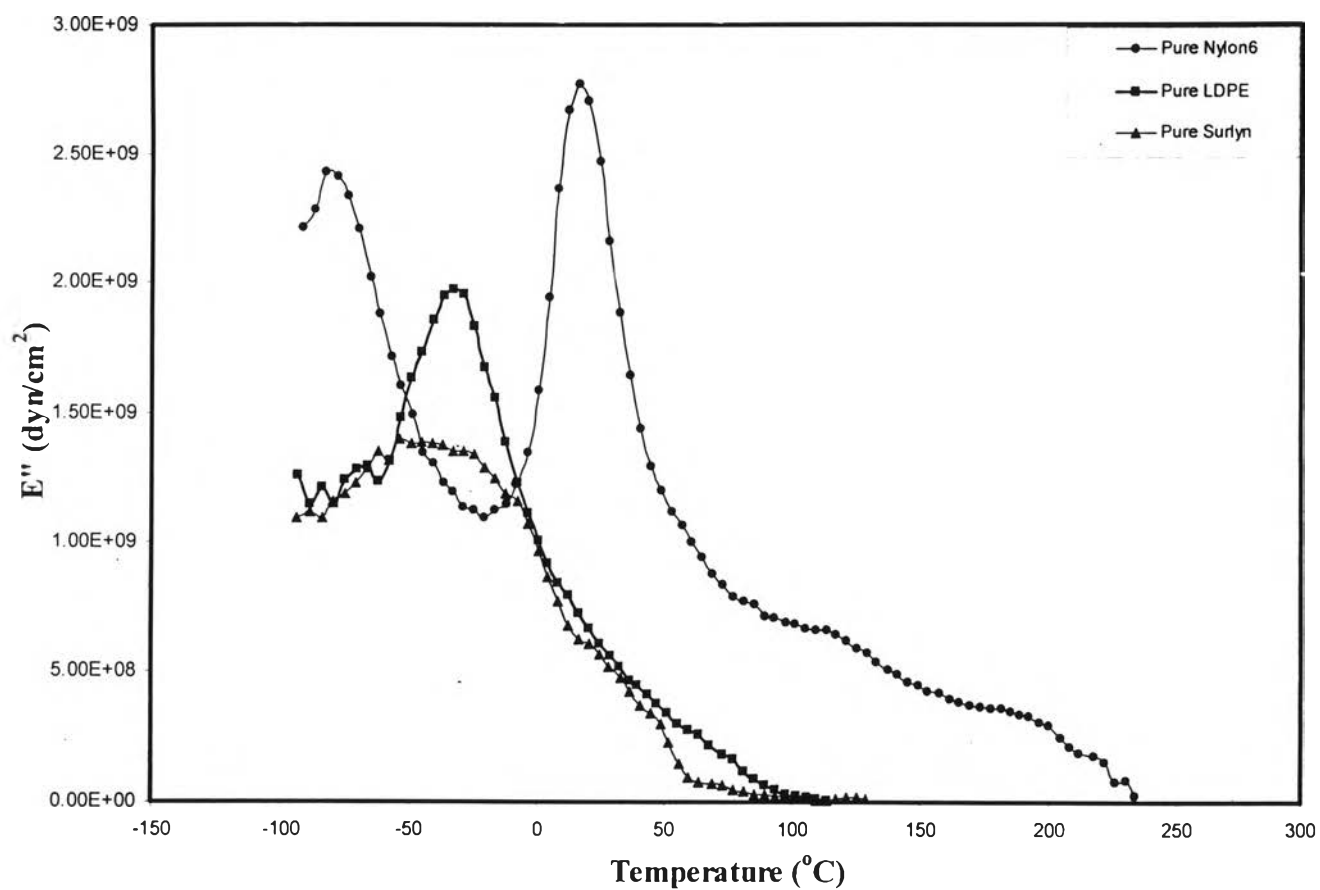


Figure 4.7 Temperature dependence of loss modulus of Pure materials: (●) Pure PA6; (■) Pure LDPE; (▲) Pure Na-EMAA ionomer.

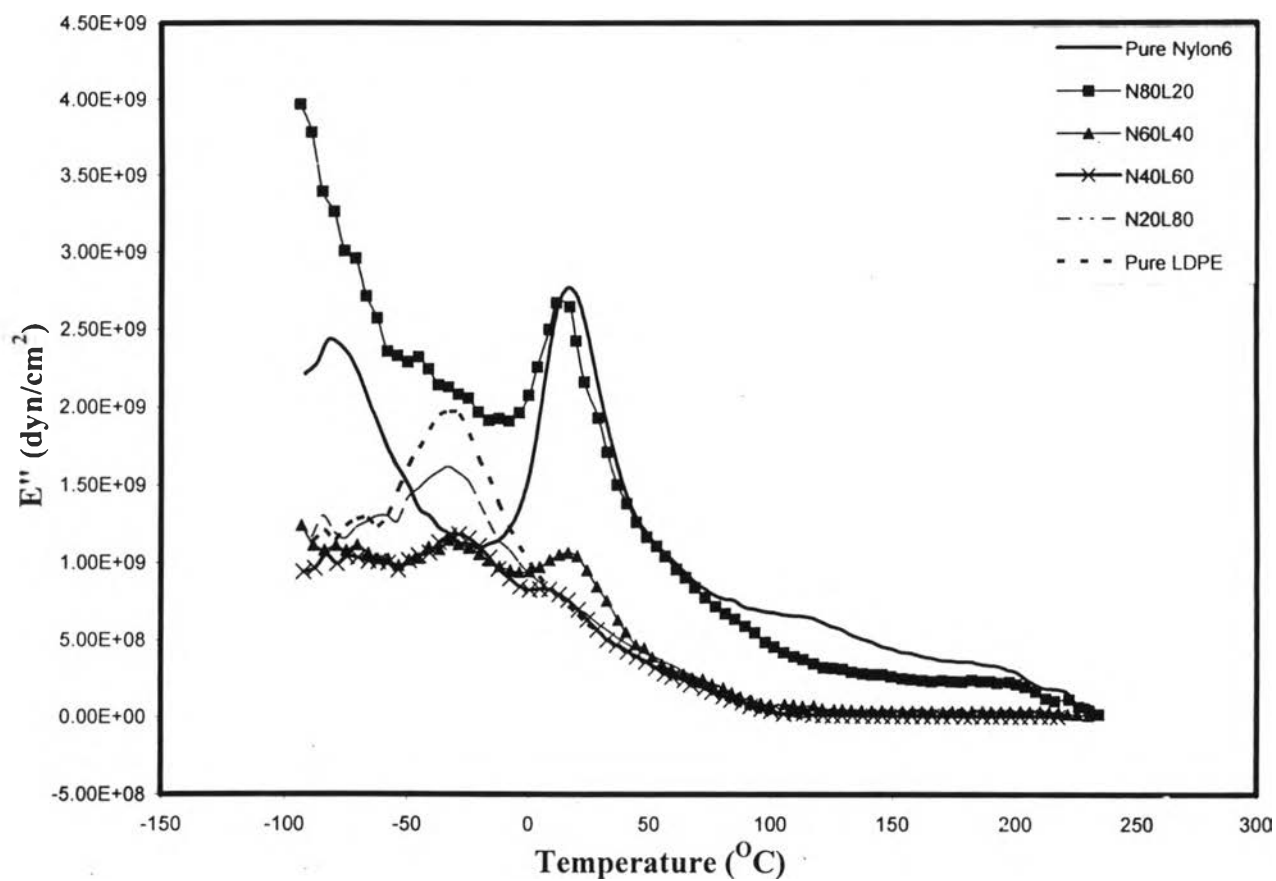


Figure 4.8 Temperature dependence of loss modulus of PA6/LDPE blends: (—) 100/0; (■) 80/20; (▲) 60/40; (×) 40/60; (---) 20/80; (---) 0/100.

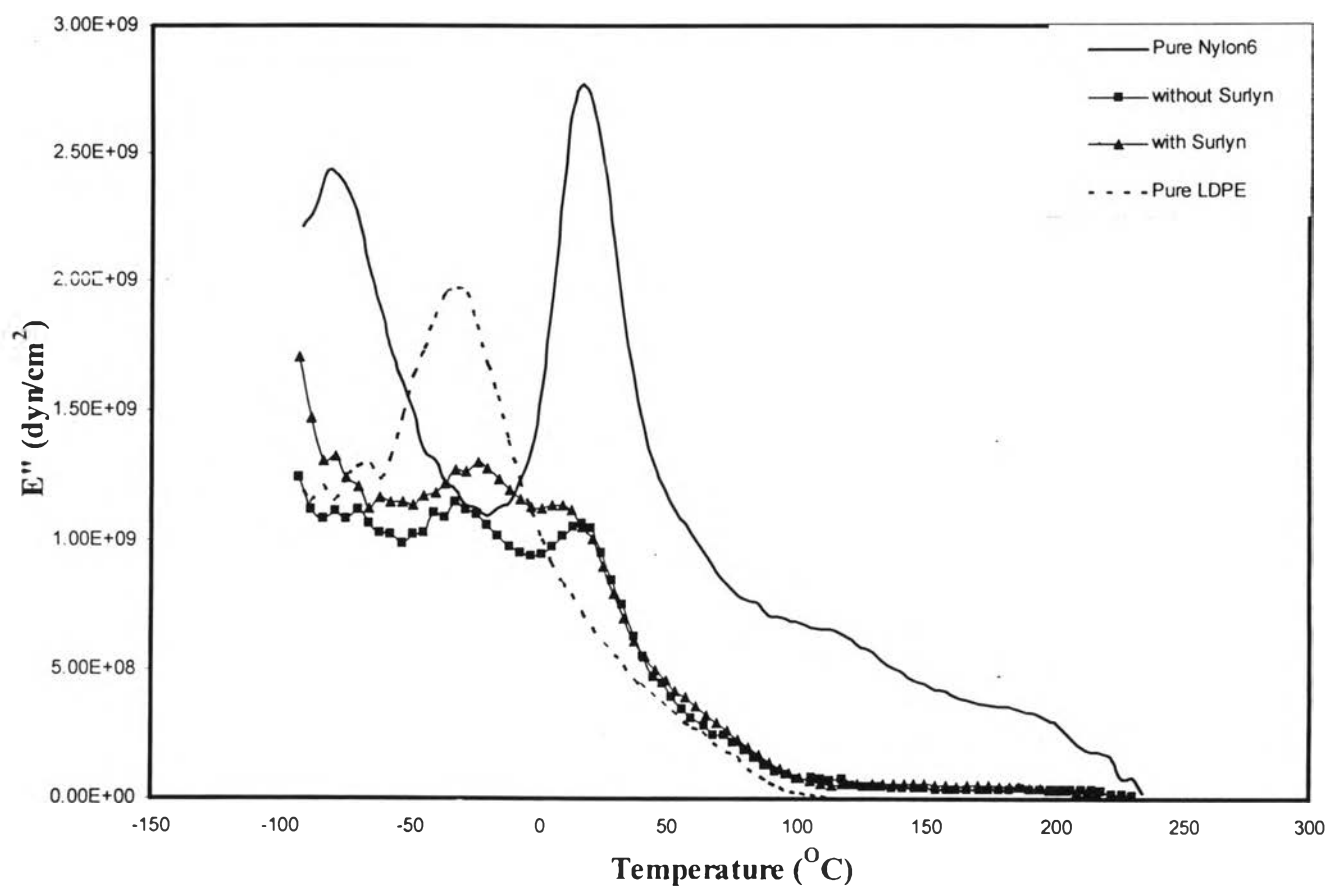


Figure 4.9 Temperature dependence of loss modulus of PA6/LDPE blends with and without compatibilizer.

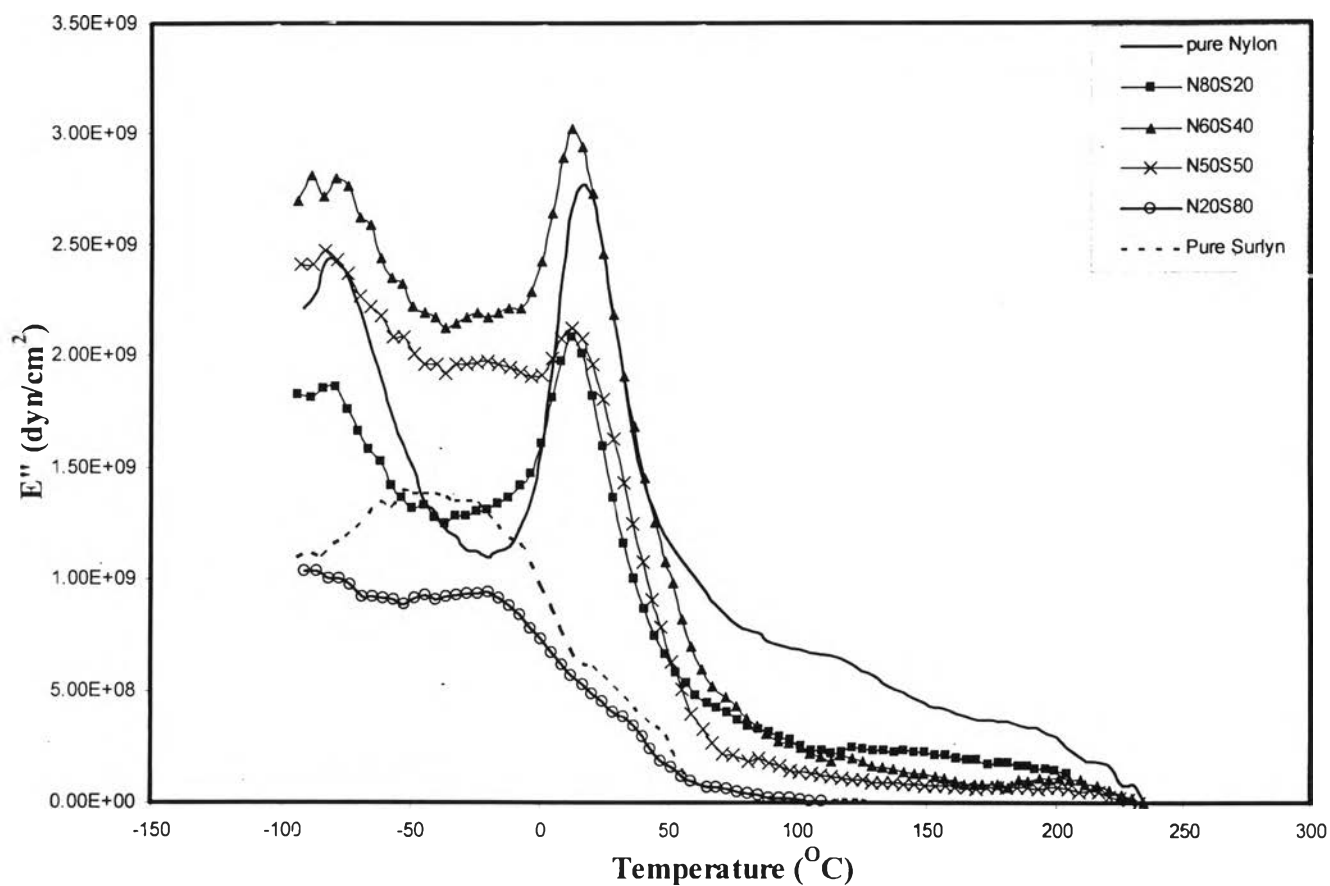


Figure 4.10 Temperature dependence of loss modulus of PA6/Na-EMAA ionomer blends: (—) 100/0; (■) 80/20; (▲) 60/40; (×) 50/50; (○) 20/80; (---) 0/100.

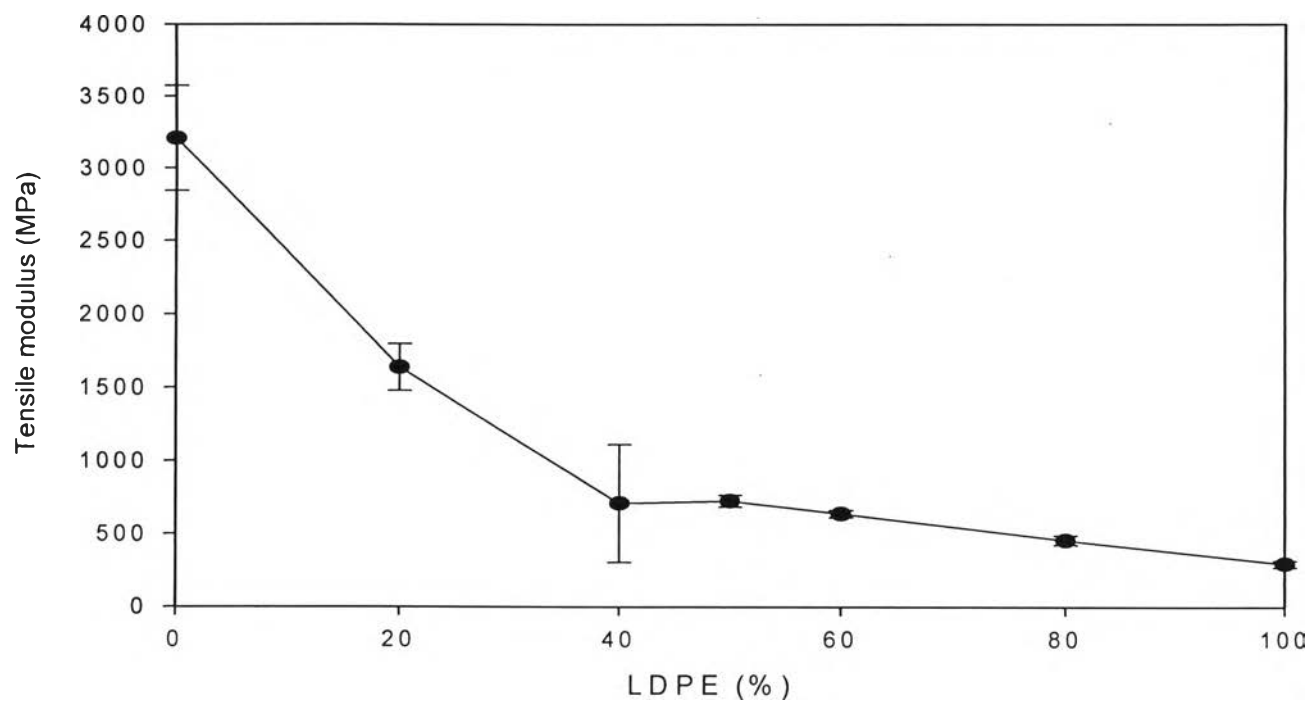


Figure 4.11 Tensile modulus of uncompatibilized PA6/LDPE blends.

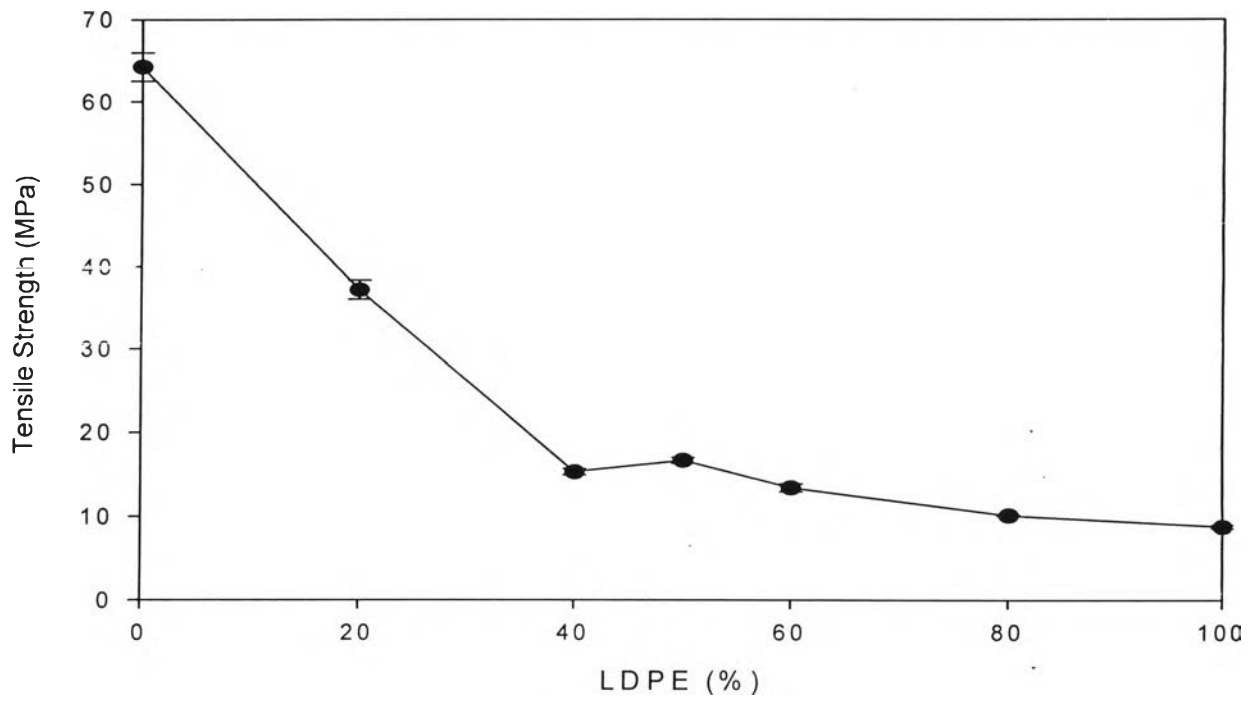


Figure 4.12 Tensile strength of uncompatibilized PA6/LDPE blends.

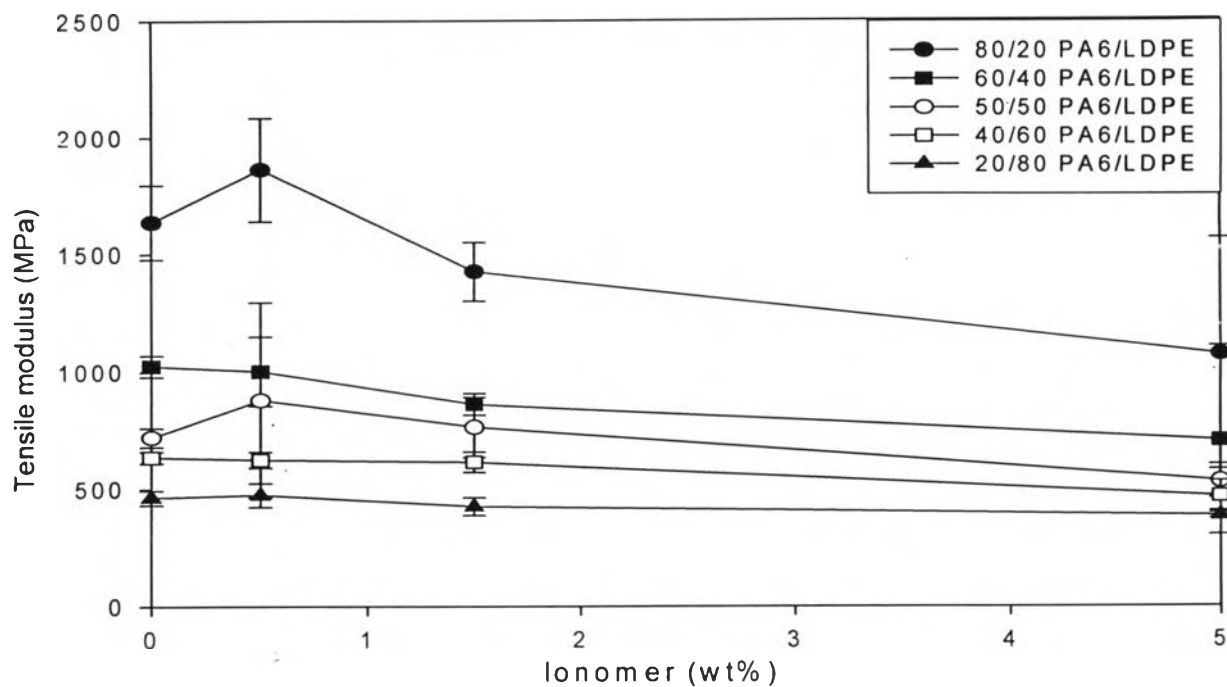


Figure 4.13 Tensile modulus of PA6/LDPE blends as a function of Na-EMAA ionomer (Surlyn[®]) content.

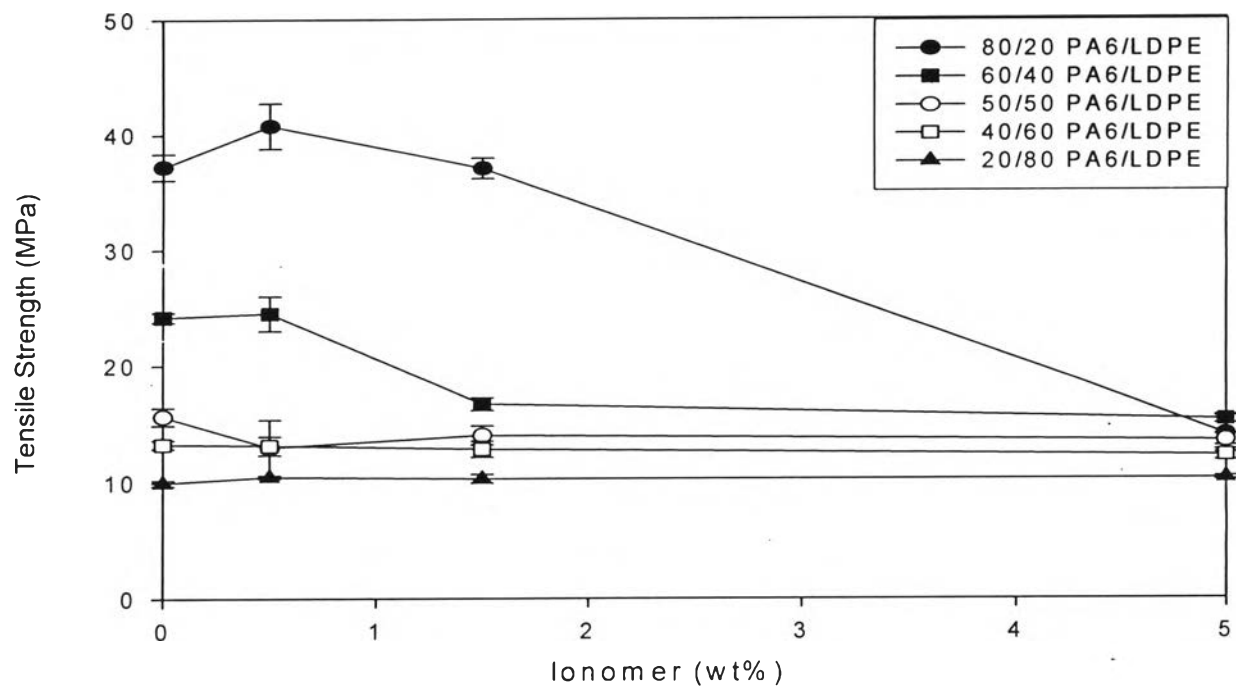


Figure 4.14 Tensile strength of PA6/LDPE blends as a function of Na-EMAA ionomer (Surlyn[®]) content.

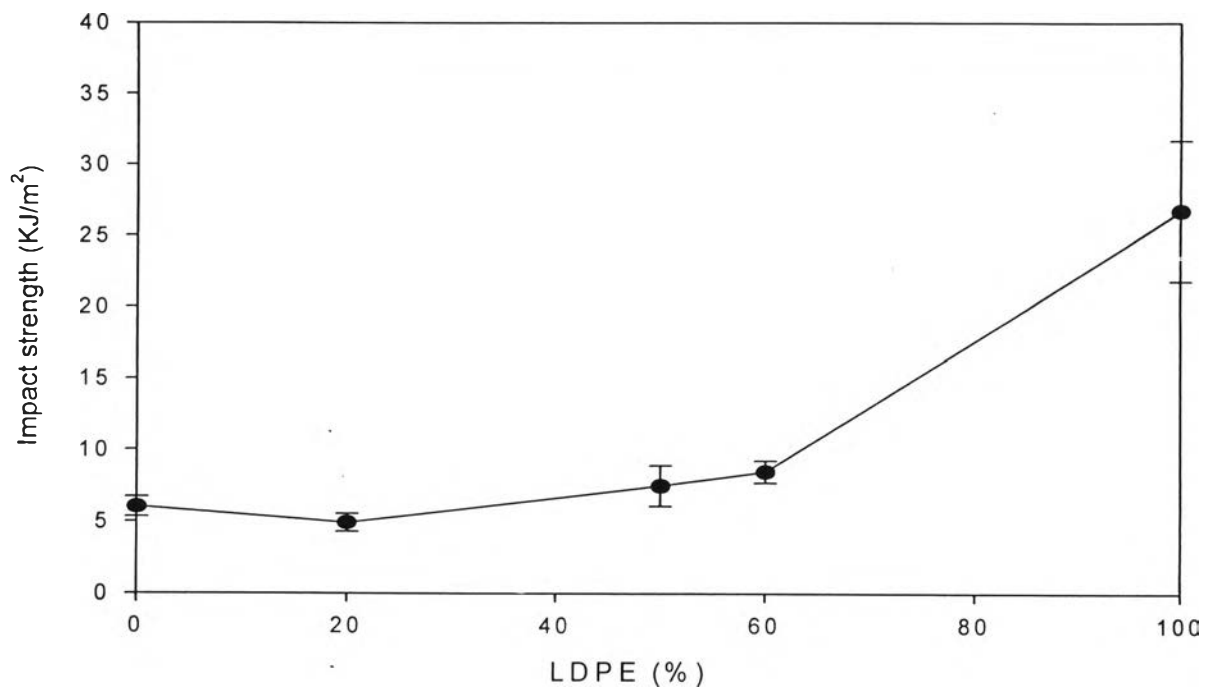


Figure 4.15 Impact strength of uncompatibilized PA6/LDPE blends.

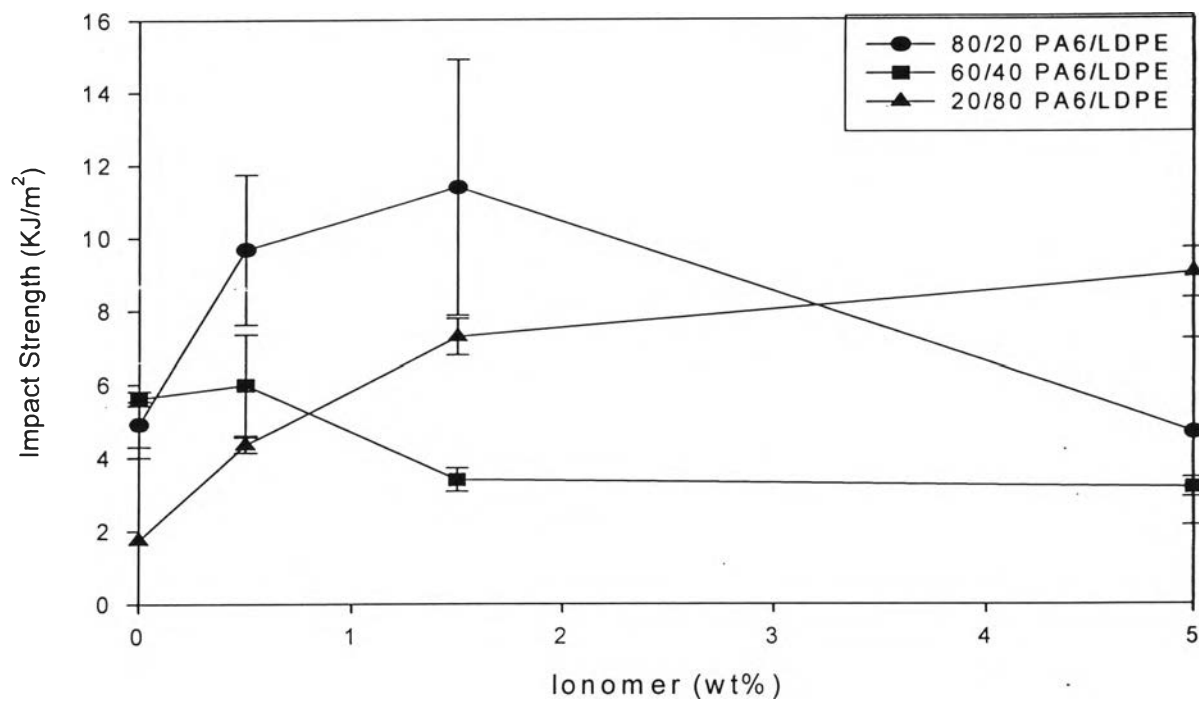


Figure 4.16 Impact strength of PA6/LDPE blends as a function of Na-EMAA ionomer (Surlyn[®]) content.

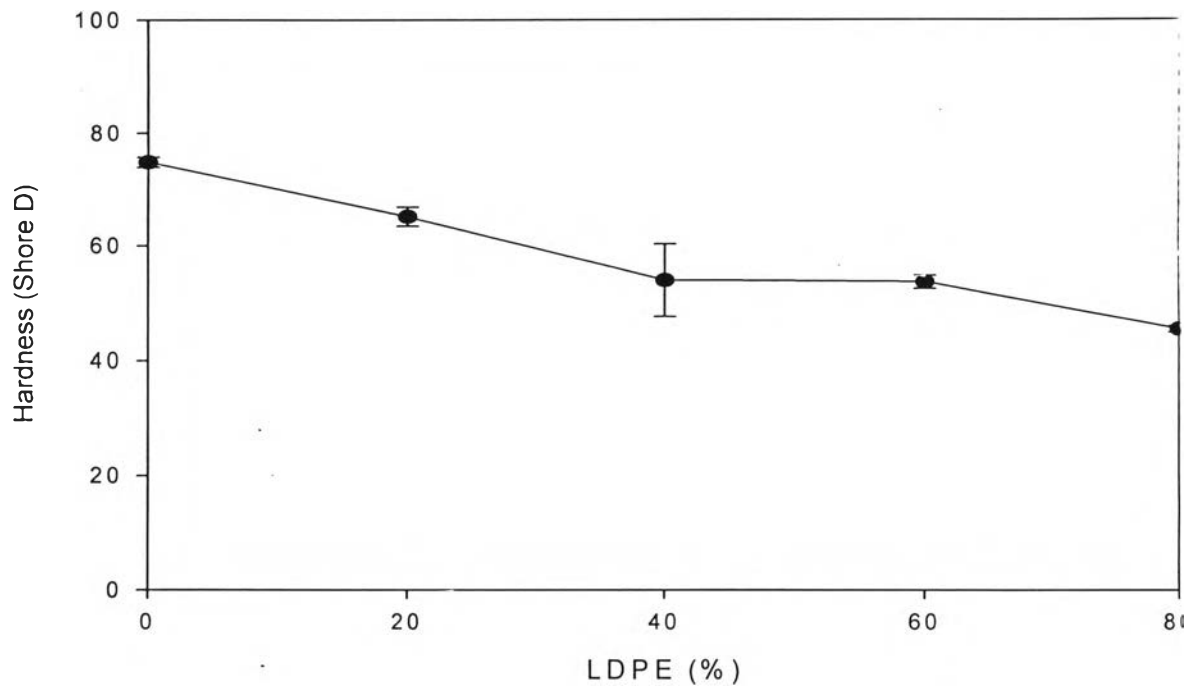


Figure 4.17 Hardness of uncompatibilized PA6/LDPE blends.

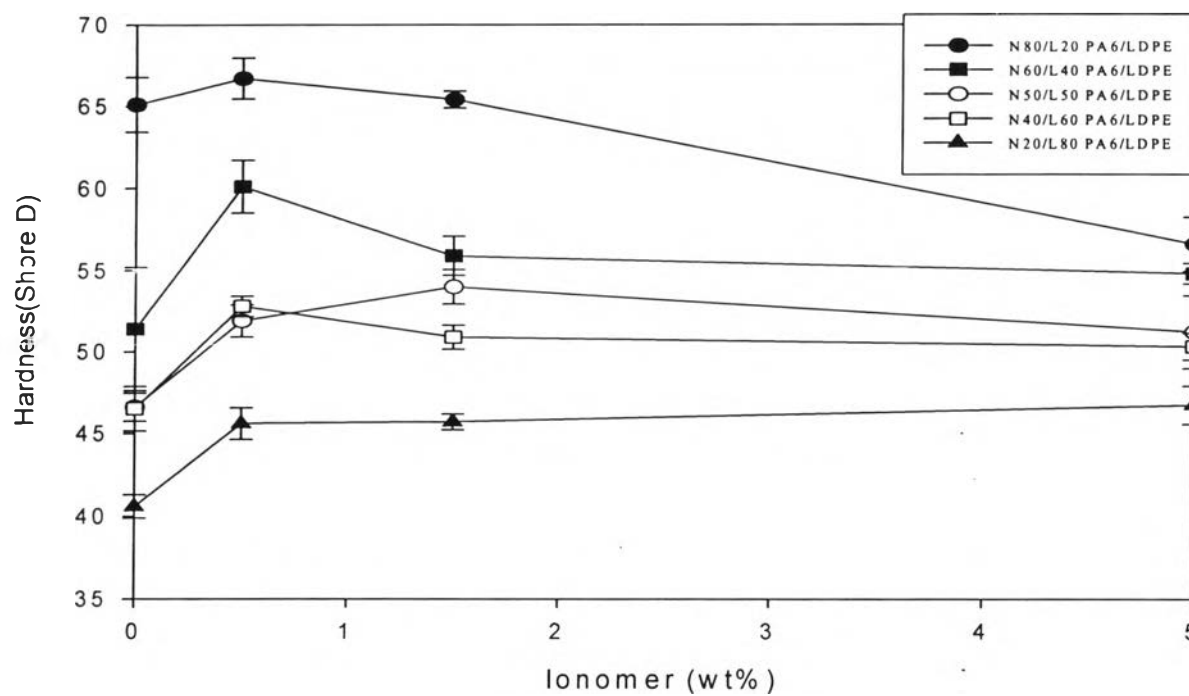


Figure 4.18 Hardness of PA6/LDPE blends as a function of Na-EMAA ionomer (Surlyn®) content.

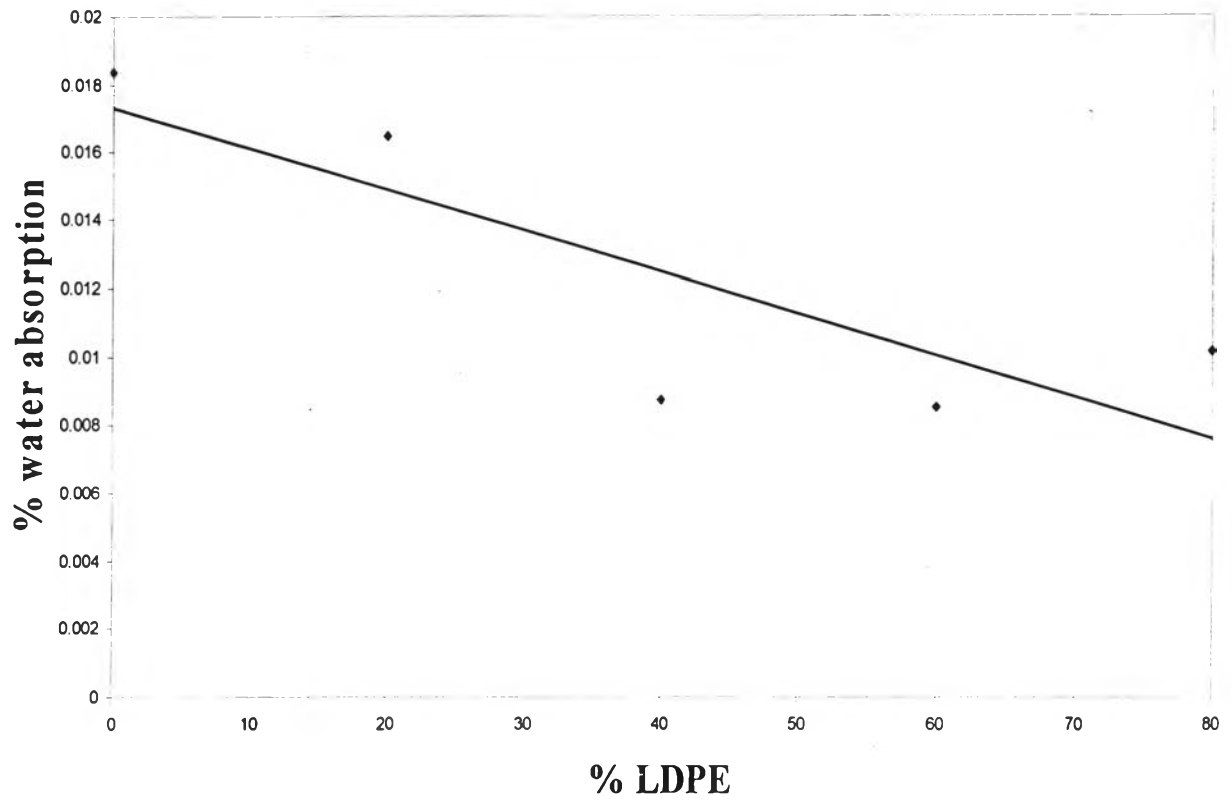


Figure 4.19 The percentage water absorption of uncompatibilized PA6/LDPE blends.

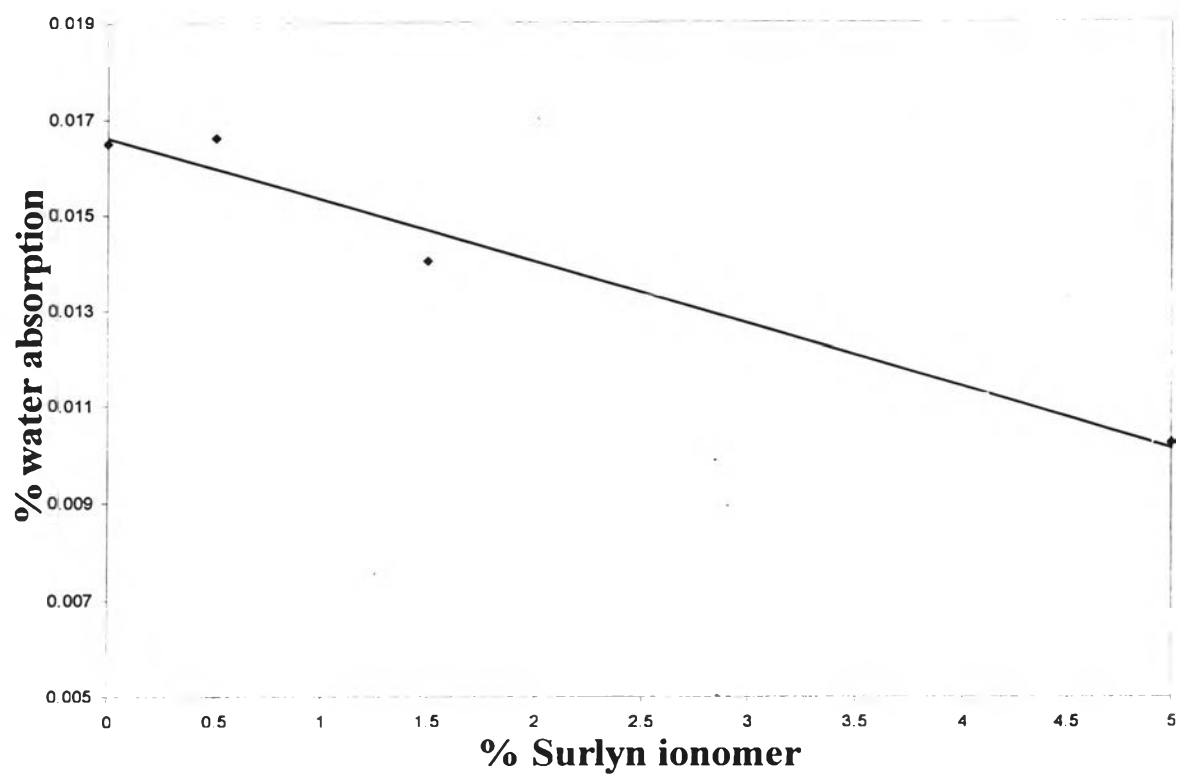


Figure 4.20 The percentage water absorption of PA6/LDPE blends as a function of Na-EMAA ionomer (Surlyn[®]) content.

Table 4.1 Number average diameter of dispersed phase size of blends

Blend type Ratio (%wt)	Number average diameter (μm)				
	20/80	40/60	50/50	60/40	80/20
PA6/LDPE	13.4	12.3	13.3	13.1	15.3
PA6/LDPE with 0.5 phr Na-EMAA	2.4	2.7	5.1	2.3	2.2
PA6/LDPE with 1.5 phr Na-EMAA	1.6	2.2	2.3	2.1	1.6
PA6/LDPE with 5 phr Na-EMAA	2.4	1.9	1.6	1.4	1.5
PA6/ Na-EMAA	1.2	1.8	1.8	1.9	0.9